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PHYSICAL CHARACTERIZATION OF ELECTRONIC MATERIALS, DEVICES AND THIN FILMS

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13. ABSTRACT

In support of research being conducted by the Properties and Phenomena Branch, Solid State Sciences Laboratory, Air Force Cambridge Research Laboratory, ManLabs, Inc. is conducting a service effort that is directed toward the characterization of specified physical, chemical and structural properties of various materials. Experimental methods include chemical analysis, reflection electron microscopy and diffraction, X-Ray diffraction and flourescence analysis, light microscopy and electron microprobe analysis, in addition to the determination of specific properties, such as density, hardness and thermal conductivity. Special services, such as crystal orientation, cutting, grinding and polishing are also being performed. Specific materials submitted for characterization include spinel, lithium germanate, silicon germanium, silicon carbide, quartz, ruby, gallium arsenide, yttrium-iron garnet, yttrium-aluminum garnet, lithium niobate, bismuth germanate, rubidium iodate, lithium tantalate, sodium chloride, calcium fluoride, zirconium, cadmium teluride, potassium chloride, cobalt silicide, β-silicon carbide, gallium phosphide, Nd-glass, and rubidium bromide. In addition, a variety of specimens have been submitted for specific studies such as phase identification, crystallinity and chemical analysis.

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FOREWORD

This report was prepared by ManLabs, Inc., 21 Erie Street Cambridge, Massachusetts, under Air Force Contract No. F19628-70-C-0140. The work was administered by the Office of Aerospace Research, Air Force Cambridge Research Laboratories, United States Air Force, Bedford, Massachusetts, with Mr. Norman Pickering providing technical liaison.

ManLabs personnel that have participated in this program are Dr. S. Andrew Kulin, Dr. Edward V. Clougherty, Konstantin Kreder Harvey Nesor, Edward P. Warekois, Joseph Davis, Kathleen Meany, Henry Plimpton.

This is a technical report and describes studies which were initiated 1 December 1970 and were concluded 1 December 1971.

ABSTRACT

In support of research being conducted by the Properties and Phenomena Branch, Solid State Sciences Laboratory, Air Force Cambridge Research Laboratory, ManLabs, Inc. is conducting a service effort that is directed toward the characterization of specified physical, chemical and structural properties of various materials. Experimental methods include chemical analysis, reflection electron microscopy and diffraction, X-Ray diffraction and flourescence analysis, light microscopy and electron microprobe analysis, in addition to the determination of specific properties, such as density, hardness and thermal conductivity. Special services, such as crystal orientation, cutting, grinding and polishing are also being performed. Specific materials submitted for characterization include spinel, lithium germanate, silicon germanium, silicon carbide, quartz, ruby, gallium arsenide, yttrium-iron garnet, yttrium-aluminum garnet, lithium niobate, bismuth germanate, rubidium iodate, lithium tantalate, sodium chloride, calcium fluoride, zirconium, cadmium teluride, potassium chloride, cobalt silicide, β-silicon carbide, gallium phosphide, Nd-glass, and rubidium bromide. In addition, a variety of specimens have been submitted for specific studies such as phase identification, crystallinity and chemical analysis.

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I. INTRODUCTION

ManLabs, Inc. is conducting a service and research effort in support of work carried out by the Properties and Phenomena Branch, Solid State Sciences Laboratory, AFCRL. This effort is directed to the characterization of the physical properties of electronic materials, devices and thin films and to the fabrication of various electro-optical experimental components. The experimental methods which are employed have been described in "Physical Characterization of Electronic Materials, Devices and Thin Films", by S. Andrew Kulin and Konstantin Kreder, Scientific Report No. 1, AFCRL Contract No. F19628-70-C-0140, January 1, 1971.

Requests for services are individually submitted and experimental results are conveyed in letter reports. Liaison personnel are Jane Bruce and Norman Pickering, AFCRL and Dr. S. Andrew Kulin and K. Kreder, ManLabs, Inc.

Personnel contributing to the materials characterization studies and services during this reporting period include Dr. S. A. Kulin, Harvey Nesor, Konstantin Kreder, Joseph Davis, E.P. Warekois, K. Meaney and Harry Tushman.

II. EXPERIMENTAL METHODS

The following experimental methods and procedures have been employed in the course of the program.

A. X-ray Diffraction

Back reflection X-ray patterns have been utilized to determine the crystallinity of specimens. Polycrystalline materials yield ring (pin hole) patterns which can be used for qualitative measures of grain size, lattice parameter and preferred orientation. Single crystal materials yield spot (Laue) patterns which are utilized to confirm crystallinity, to provide crystal orientation and to give a qualitative estimate of crystal strain and perfection. The lattice perfection is based upon observed spot distortion, diffuseness or fragmentation. Single crystal orientation is carried out by conventional back-reflection Laue methods. The specimen holder is transferred from the X-ray unit directly to the grinding machine where specific specimen configurations are prepared by appropriate cutting and grinding. When required, the specimen faces are polished by hand lapping with diamond, alumina, or other special abrasive materials.

Quantitative crystal perfection measurements have been carried out by means of a double crystal spectrometer. In operation, an analysing crystal of high perfection is used to produce a monochromatic, parallel X-ray beam. This radiation impinges upon the second (specimen) crystal, which is slowly rotated through an angle ω about a rocking axis which is included in the surface of the specimen and is perpendicular to the X-ray plane. The resulting rocking curve (X-ray intensity versus ω) that is obtained from reflection planes parallel to the specimen surface has an

angular breadth at half maximum intensity which is directly related to small angle tilt boundaries and dislocation-strain distributions within the specimen. After correcting for geometrical and natural (Darwin-Prins) broadening, the half breadth values can be utilized in calculating dislocation densities.

B. Crystal Cutting and Polishing

The experimental procedures used to fabricate crystals of various sizes and shapes were generally as follows: The boule was mounted onto a brass block with "quartz cement" and was X-ray oriented to the specific crystallographic plane. The required shapes were then cut and ground with diamond wheels using a Sanford Surface Grinder and machine polished or lapped using a Crane Lapmaster 12. After rough polishing, the final orientation of the crystal was checked by taking an X-ray Laue pattern and in certain cases, by use of a horizontal diffractometer.

In order to obtain crystals of good quality, i.e. smooth surface and completely without flaws, the machine lapping operation was followed by hand lapping on a precision ground glass covered with suitable abrasive and moving with a sweeping motion over the glass surface preferably in the form of figure eights. Surface flatness of about one wavelength of sodium light and surface parallelism of about 10 sec. of arc or better was routinely maintained during the fabrication of the crystallographically oriented plates of quartz, linobates, germanates and other crystals.

C. Electron Diffraction

The crystallinity and orientation of several thin film deposits have been examined by reflection electron diffraction in which the specimen is placed almost parallel to an electron beam. Electrons diffract from surface asperities and are recorded on a film placed below the specimen.

Amorphous materials yield very broad, diffuse ring or halo patterns, polycrystalline materials yield concentric ring patterns and single crystal materials yield spot patterns. The ring patterns can be utilized to determine interplanar spacings of the reflecting planes, allowing specimen indentification. Single crystal spot patterns can be analyzed to determine both the structural identification and orientation of the specimen.

Particular orientations are achieved by rotations about two orthogonal axes normal to the electron beam and by a lateral translation. The camera constant (i.e., the relation between ring radius or spot distance from film center to interplaner spacing) is determined by means of a known standard. This constant is strongly dependent on electron beam voltage, but is rather insensitive to other microscope adjustments. All examinations reported herein have been carried out on a Hitachi HU-11 Electron Microscope operated at 100kv.

D. Chemical Analyses

A fully equipped analytical laboratory was available for wet chemical, emission spectrographic and spectrochemical analyses.

E. Hardness

Specimens were mounted in epoxy and were polished to about a 1 micron surface finish to provide a uniform flat surface. Hardness measurements were made on a Leitz Durimet Microhardness Tester utilizing either Knoop or Vickers pyramid indentors. Hardness values are reported in kg/mm^2.

III. EXPERIMENTAL RESULTS

A listing of the various services provided during the past quarter is presented in Table 1. The experimental findings are summarized below.

A. X-Ray Procedures - Crystal Orientation

The orientation of a number of crystals was determined using back-reflection X-ray Laue techniques. Laue patterns were also utilized to indicate crystal perfection. When a precision crystallographic orientation was required, the crystal was first X-ray oriented within $\pm 1/2^{\circ}$ with respect to a given crystallographic direction using a Laue Back Reflection Camera after which a set of reference surfaces was optimized using a Philips Horizontal X-Ray Diffractometer with independent movement of the θ and 2θ arms. The precision of crystal orientation by this method is known to be at least $\pm$ 5 minutes of arc. The results of the crystal orientation work is summarized in Table 2.

TABLE 1

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen No.	Material	Analytical Methods*
4-21	04116	Pitha	G-1-274	GaAs	CC
4-22	04053	Szabo	11-25-70B	$LiNbO_3$	CC
4-23	04115	Pitha		Germanium	CC
4-24	04054	Slobodnik	12-8-70	GaAs	XRL, CC
			12-19-69	GaAs	CC
5-1	04150	Pitha		Germanium	CC
5-2	04055	Slobodnik	7-27-70	$Bi_{12}GeO_{20}$	CC
			1-5-71	$Bi_{12}GeO_{20}$	CC
5-3	04056	Slobodnik	12-14-70	$LiNbO_3$	CC
5-4	04057	Slobodnik	12-17-70	$Bi_{12}GeO_{20}$	CC
5-5	04058	Carr		$LiNbO_3$	CC
5-6	04059	Slobodnik	AA-34	$Bi_{12}GeO_{20}$	XRL, XRD
5-7	04060	Slobodnik	1-5-71	$Bi_{12}GeO_{20}$	CC
5-8	04106	Posen	RB-1	$RbIO_3$	CA

*XRL-X-ray Laue, XRD-X-ray diffraction analysis, CC-crystal cutting and / or grinding, polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy: EPM-electron probe microanalysis; D-density; TE-thermal expansion.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requester	Specimen No.	Material	Analytical Method*
5-9	04176	Pitha	G-74A	Quartz	CC
			G-74B	Quartz	CC
			G-69	Quartz	CC
			G-47	Quartz	CC
5-10	04061	Slobodnik	11-22-70	$Bi_{12}GeO_{20}$	CC
5-11	04062	Slobodnik	8-19-70	$LiTaO_3$	XRL, XRD, CC
5-12	04142	Hilton	11-25-69	$LiNbO_3$	CC
			12-10-69	$LiNbO_3$	CC
5-14	04064	Slobodnik	11-25-70	$Bi_{12}GeO_{20}$	CC
5-15	04211	Brown	C2-1	Quartz	XRL, CC
5-16	04189	Pitha	30^{o}Wedge	Sapphire	XRL, CC
			50^{o}Wedge	Sapphire	XRL, CC
5-17	04065	Slobodnik	7-27-70	$Bi_{12}GeO_{20}$	CC
			1-5-71	$Bi_{12}GeO_{20}$	CC

*XRL-X-ray Laue XRD-X-ray diffraction analysis, CC-crystal cutting and / or grinding, polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen No.	Material	Analytical Methods*
5-18	03175	Pitha	Disc	Quartz	CC
5-19	03176	Lipson	Disc	NaCl	CC
5-20	04159	Smiltons	JS-Zr	Zirconium	CA
5-21	04066	Slobodnik	2-12-71	$Bi_{12}GeO_{20}$	CC
5-22	04067	Szabo	KE-6A	$LiNbO_3$	CC
			11-25-70B	$LiNbO_3$	CC
5-23	03193	Pitha	Disc	Quartz	CC
5-24	03194	Lipson	Disc No. 1	NaCl	CC
			Disc No. 2	NaCl	CC
			Disc No. 3	NaCl	CC
			Disc No. 4	NaCl	CC
5-25	03195	Lipson	Plate No. 1	CaF_2	CC
			Plate No. 2	CaF_2	CC

*XRL-X-ray Laue, XRD-X-ray diffraction analysis, CC-crystal cutting and / or grinding, polishing; CA-chemical analysis; H-hardness; ES emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen No.	Material	Analytical Methods **
6-1	04068	Slobodnik	2-24-71	$LiNbO_3$	CC
			2-22-71	BGO	CC
6-2	03210	Lipson		NaCl	CC
6-3	04069	Slobodnik	3-5-71	BGO	CC
6-4	04070	Slobodnik	12-18-69	GaAs	XRL, XRD
6-5*	04077	Slobodnik	BeO-SA1	BeO	XRL, XRD, CC
6-6	04078	Slobodnik	7-27-70T	BGO	CC
6-9	04079	Carr	ST-Cut	Quartz	CC
6-10	04080	Slobodnik	8-14-70	Quartz	CC
6-11	04081	Slobodnik	3-1-71D	BGO	CC
			4-1-71S	BGO	CC
6-13	04083	Slobodnik	4-27-71	$LiNbO_3$	CC
			[illegible]-30-71	BGO	CC
6-14	04084	Carr	S[illegible]-Cut	Quratz	CC

*Work in progress

**XRL-X-ray Laue, XRD-X-ray diffraction analysis, CC-crystal cutting and/or grinding, polishing; CA-chemical analysis; H-Hardness; ES-emission spectroscopy: EPM-electron probe microanalysis; D-density; TE-thermal expansion; F-Fabrication; ED-Electron Diffraction.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen No.	Material	Analytical Method**
6-15	04085	Slobodnik	--	$LiTaO_3$	XRL-XRD
6-16	04216	Capone	A6-17	Quartz	XRL, XRD, CC
6-17	04029	Posen	LQ-11	CdTe	CC
6-18	04404	Pitha		KCl	XRL, CC
6-19	04086	Slobodnik	5-21-71	$LiNbO_3$	XRL, XRD
			3-15-71	$LiNbO_3$	CC
6-20	02881	Brown		Quartz	XRL, CC
6-21	04441	Bruce	No. 7	KCl	CC
			No. 8	KCl	CC
			No. 9	KCl	CC

*Work in progress

**XRL-X-ray Laue; XRD-X-ray diffraction analysis, CC-crystal cutting and/or grinding, polishing; CA-chemical analysis: H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen No.	Material	Analytical Methods**
7-1	02884	Brown	-------	Quartz	XRL, CC
7-2	02883	Brown	SWEPT	Quartz	XRL, CC
7-3	04442	Lipson	LQ15	KCl	XRL, F, CC
7-4	04443	Lipson	--------	NaCl	F, CC
7-5	02594	Berman	91A, 91B, 91C 71	CoSi, β SiC CoSi β SiC	ED
7-6	03000	Lipson	6-1-1-71	BGO-Nd	F, CC
7-7	04087	Carr	------	YAG	CC
7-8	02597	Berman	90, 92, 93, 94	β SiC and CoSi	ED
7-9	04088	Slobodnik	3-5-71T	BGO,	CC
7-10	04475	Lipson	-------	NaCl	CC

*Work in Progress

**XRL-X-ray Laue: XRD-X-ray diffraction analysis, CC-crystal cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES emission spectroscopy; EPM-electron probe microanalysis; D-density; TE thermal expansion; F-fabrication; ED - electron diffraction.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFRCL No.	Requestor	Specimen Nos.	Material	Analytical Methods**
7-11	04089	Solobodnik	12-21-1-70 6-28-71 6-29-71	$LiTaO_3$ $LiTaO_3$ $LiTaO_3$	XRL, XRD, CC
7-12	04091	Slobodnik	4-30-71	BGO	CC
7-13	04478	Capone	------	Quartz	XRL, XRD, F, CC
7-14	04479	Lipson	------	NaCl	CC
7-15	04092	Slobodnik	26187	$LiNbO_3$	XRL
7-16	02888	Brown	1700-6, 261-22, A6-17 C3-80 EG	Quartz	
7-17	02889	Brown	C2-1	Quartz	XRL, F
7-18	03025	Lipson	------	NaCl	CC
7-19	02693	Berman	100B, 103A 104B, 105 98, 104A 100A, 105B	β SiC	ED
7-20	04453	Lipson	------	NaCl	CC
7-21	02694	Berman	107A, 107P, 108A 108P	β SiC	ED
7-22	04093	Slobodnik	3-5-71T 3-27-70T	BGO	CC
7-23	04090	Sethares	------	Ga-YIG	CC
7-24	04094	Carr	------	YAG	CC

*Work in Progress

** XRL-X-ray Laue, XRD-X-ray diffraction analysis, CC-crystal cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion; F-fabrication; ED electron diffraction.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen Nos.	Material	Analytical Methods**
7-25	03004	Kennedy	------	GaAs	CC
7-26	02891	Brown	E-9	Quartz	XRL, F, CC
7-27	04445	Armington	Ga P-1	GaP	XRL, CC
7-28	03030	Lipson	TI-20	Nd-Glass	F, CC
7-29	04447	Kimerling	-----	Si	XRL, CC
7-30*	04491	Wiener	------	Cd-Te	CA
7-31	04095	Slobodnik	12-19-69 12-8-70 3-27-71	GaAs	CC
7-32	04096	Slobodnik	5-21-71	$LiNbO_3$	XRL

*Work in Progress

** XRL-Xray Laue, XRD-X-Ray diffraction analysis, CC-crystal cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion; F-fabrication; ED-electron diffraction.

TABLE 1 (Cont)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen Nos.	Material	Analytical Methods**
8-1	03042	Lipson	LQ-50	KCl	XRL, F, CC
8-2	02696	Berman	86, 89A, 109A 109B, 111, 111B 112	SiC on Si	ED
8-3	02697	Berman	114, 115	SiC	ED
8-4	03044	Pickering	LQ-71 LQ-72	RbCl RbBr	F, CC F, CC
8-5	02893	Brown	C3-20	Quartz	XRL, F, CC
8-6	04072	Slobodnik	9-24-71	$LiNbO_3$	XRL, CC
8-7	05425	Brown	410-9-71	Quartz	XRL, F, CC
8-8	03047	Pickering	Si-3	Si	F, CC
8-9	03008	Posen	LQ-33-LQ-45 LQ-46, LQ-54	KCl	H
8-10	04073	Carr	3-15-7-	$LiNbO_3$	CC

* Work in Progress

** XRL-X-ray; XRD-X-ray diffraction analysis, CC-crystal Cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion; F-fabrication; ED - electron diffraction.

TABLE 1 (Cont.)

LIST OF ANALYTICAL SERVICES

Index	AFCRL No	Requestor	Specimen Nos.	Material	Analytical Methods**
8-11	04275	Lipson		NaCl	CC
8-12	03031	Berman	123, 123B, 124 125, 125B	SiC	ED
8-13	04074	Carr	11-11-70	Quartz	CC
8-14	05439	Brown	17006	Quartz	XRL, F, CC
8-15	05440	Brown	A10-9A	Quartz	XRL, F, CC
8-16	04496	Berman	125C125D 126A, 126B	SiC	ED
8-17	03012	Bruce	LQ-13 LQ-53 LQ-70, SQ-27 LQ-87A, LQ-88 LQ-91	RbCl K Br KCl, KBr+KCl KCl+KOH	H
8-18	04316	Pickering	LQ-34	RbBr	F, CC
8-19*	04097	Slobodnik	6-28-71	$LiTaO_3$	CC

*Work in Progress
** XRL-X-ray Laue, XRD-X-ray diffration analysis, CC-crystal cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis D-density; TE-thermal expansion; F-fabrication ED-electron diffraction.

TABLE 1 (cont.)
LIST OF ANALYTICAL SERVICES

Index	AFCRL No.	Requestor	Specimen Nos.	Material	Analytical Methods**
8-21	02698	Berman	133-2, 133-3 133-4	SiC	ED
8-22*	05441	Capone	A-10-9A	Quartz	XRL, XRD, F, CC
8-23	04099	Szabo	2-25-70	$LiNbO_3$	CC
8-24	04098	Slobodnik	10-27-71	$LiNbO_3$	CC
8-25	05438	Capone	AF-10P-010-LPG	Si	XRL, F
8-26*	05429	Armington	LQ-92	GAP	XRL, F, CC
8-27*	05442	Buckmelter	10-20-1-71	GAP	F, CC
8-28*	05443	Clark		NaCl	CC

*Work in Progress

**XRL-X-ray Laue, XRD-X-ray diffraction analysis, CC-crystal cutting and/or grinding polishing; CA-chemical analysis; H-hardness; ES-emission spectroscopy; EPM-electron probe microanalysis; D-density; TE-thermal expansion; F-fabrication; ED-electron diffraction.

TABLE 2

CRYSTAL ORIENTATION

Job No.	Spec. No.	Description	Orientation hkl
04059	AA-34	$Bi_{12}GeO_{20}$	(110)
04062	8-19-70	$LiTaO_3$	(0001), $(10\bar{1}0)$
04211	C2-1	Quartz	(0001), $(10\bar{1}0)$, $(2\bar{1}\bar{1}0)$
04189	30° Wedge	Sapphire	(0001), $(10\bar{1}0)$
	50° Wedge	Sapphire	(0001), $(10\bar{1}0)$
04070	12-18-69	GaAs	(111), (110), (112)
04077	BeO-SA1	BeO	$(10\bar{1}0)$, (0001), $(2\bar{1}\bar{1}0)$
04075		BGO	(110), $(1\bar{1}0)$, (001)
04076		$LiNbO_3$	$(10\bar{1}0)$
04082		Quartz	(0001), $(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$
04085		$LiTaO_2$	(0001), $(10\bar{1}0)$
04216	A6-17	Quartz	AT-Cut
04404		KCl	(001), (010), (100)
02881		Quartz	(0001)
02884		Quartz	$(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$, (0001)
02883	SWEPT	Quartz	$(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$, (0001)
04442	LQ 15	KCl	(100), (010), (001)
04089	6-28-71	$LiTaO_3$	$(10\bar{1}0)$, (0001)
04478		Quartz	$(10\bar{1}1)$ AT-Cut
03092	26187	$LiNbO_3$	$(10\bar{1}0)$, (0001), plate Normal 20° off $(10\bar{1}0)$
02888	1700-6, 261-22 A6-17, C3-80, EG	Quartz	$(10\bar{1}0)$
02889	C2-1	Quartz	$(10\bar{1}0)$

TABLE 2 (Cont)

CRYSTAL ORIENTATION

Job No.	Spec. No.	Description	Orientation, hkl
02891	E-9	Quartz	$(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$, (0001)
04445	Ga P-1	GaP	(111)
04447		Si	(110)
04096	5-21-71	$LiNbO_3$	$(10\bar{1}0)$, (0001), plate Normal 20° from $(10\bar{1}0)$
02893	C3-20	Quartz	(0001), $(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$
05425	A10-9-71	Quartz	$(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$
05439	1700-6	Quartz	$(10\bar{1}0)$
05440	A10-9A	Quartz	(0001), $(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$
04333	A6-17 A10-9A C2-1 C3-80 261-22 1700-6	Quartz	(0001), $(2\bar{1}\bar{1}0)$, $(10\bar{1}0)$
05441	A10-9A	Quartz	Plate Normal 35°21' off (0001)
05438	AF10-P-01 LPG	Silicon	(100), (110)
05429	LQ-92	GAP	(111)

B. Crystal Cutting and Polishing

A number of quartz, YIG, YAG, potassium chloride, sodium chloride, gallium arsenide, gallium phosphide, bismuth germanate, lithium niobate and lithium tantalate crystals have been X-ray oriented, cut and polished according to stated size specifications and specimen orientations. The experimental procedures were generally as follows: the crystals were mounted onto steel blocks with "quartz cement" and were oriented to the specified crystal plane by back reflection X-ray Laue methods. The required pieces were fabricated by cutting and grinding with diamond wheels and were then polished to obtain the desired surface finish. The final orientations were checked by X-ray Laue patterns and, in certain cases, by use of a horizontal diffractometer designed for single crystal orientation work

A number of services requests during this reporting period required crystals to be polished having surface finishes at least $\lambda/10$ in flatness.

The various crystals and specimen dimensions are given in Table 3.

TABLE 3

CRYSTAL CUTTING: CRYSTAL IDENTIFICATION AND SPECIMEN REQUIREMENTS

Job No.	Spec. No.	Crystal Type	Dimensions
04116	G-1-274	GaAs	20° wedge
04115		Germanium	10° wedge
04150		Germanium	5/8" diam x 3/16" thick disc 5/8" diam x 3/16" thick disc
04176	G-74A G-74B G-69 G-47	Quartz Quartz Quartz Quartz	0.314" x 0.250" x 0.130" 0.314" x 0.250" x 1.30" 0.314" x 0.250" x 0.130" 0.314" x 0.250" x 0.130"
04211	C2-1	Quartz	1" x 1" x 1"
04189		Sapphire Sapphire	30° wedge 50° wedge
03195		CaF_2 CaF_2	5mm x 10mm x 15mm 3mm x 10mm x 15mm
04075		BGO	15mm diam x 50mm long with 5mm wide flat
04076		$LiNbO_3$	15mm diam x 5mm long with 5mm wide flat
04082		Quartz	15mm diam x 50mm long with 5mm wide flat
04216	A6-17	Quartz	15mm diam x 1.67 mm thick plate with convex surface. Four units
04029	LQ-11	CdTe	10mm diam x 1mm thick wafers. Seven units
04404		KCl	a. 1/2" x 1/2" x 1/2" cube b. 1/2" x 1/2" x 1/4" plate
02881		Quartz	3/16" diam x 2-1/4" long

TABLE 3 (Cont)

CRYSTAL CUTTING: CRYSTAL IDENTIFICATION AND SPECIMEN REQUIREMENTS

Job No.	Spec. No.	Crystal Type	Dimensions
02884		Quartz	0.575" x 0.350" x 0.080" plate 0.575" x 0.350" x 0.242" plate
02883		Quartz	0.575" x 0.350" x 0.120" plate
04442		KCl	1/2" diam x 1/4" thick cylinders (2)
04443		NaCl	3/4" diam x 1/4" thick with 5° wedge 3/4" diam x 1/4" thick with 10° wedge
03000		Nd-B60	10mm x 6mm x 4mm
04478		Quartz	15mm diam x 1.67mm height with convex surface (4)
04479		NaCl	2" diam windows (2)
02888		Quartz	0.174" diam x 2-5/16" long cylinders (5)
02889		Quartz	0.174" diam x 2-5/16" long cylinder
04090		Ga-YIG	0.800" x 0.200" x 0.050" plate
02891		Quartz	0.575" x 0.350" x 0.120" plates (5)
04445		GaP	20mm diam x 0.006" thick wafers (2)
03042	LQ-50	KCl	3/8" diam x 1" long cylinder 1/2" x 1/2" x 1" long bar
03044	LQ-71 LQ-72	RbCl RbBr	1.8cm diam x 1cm long cylinder
02893	C3-20	Quartz	0.575" x 0.450" x 0.120" plate
05425	A10-9-71	Quartz	0.575" x 0.450" x 0.120X plate (2)
03047	Si-3	Silicon	1.8cm diam x 3cm long cylinder

TABLE 3 (Cont.)

CRYSTAL CUTTING: CRYSTAL IDENTIFICATION

AND SPECIMEN REQUIREMENTS

Job No.	Spec No.	Crystal Type	Dimensions
05440	A10-9A	Quartz	1/2" x 3/4" x 1 1/2" bar
04316	LQ-34	RbBr	1.8 cm diam. x 1 cm long cylinder
04333	A6-17	Quartz	0.575" x 0.450" x 0.240" plate
	A10-9A		0.575" x 0.450" x 0.240" plate
	C2-1		0.575" x 0.450" x 0.240" plate
	C3-80		0.575" x 0.450" x 0.240" plate
	261-22		0.575" x 0.450" x 0.240" plate
	1700-6		0.575" x 0.450" x 0.240" plate
05441	A10-9A	Quartz	15 mm diam. x 1.67 mm thick plate with convex surface (4 units)
05429	LQ-92	GaP	3/16" x 3/16" x 1 1/2" long bar (4)
05442	10:20:1-71	GaP	1/8" thick sliver (3)

C. Fabrication of Modified Cylinders

During this reporting period work has progressed on the fabrication of modified cylinders out of quartz, $LiNbO_3$. amd BGO. Techniques have been applied which permit highly precise grinding of the cylinder outer diameters i.e. T.I.R. of only a few millionths of an inch. Special devices have been designed and constructed to polish the outer diameters on a centerless principle. Excellent polished surface finishes have been obtained.

The fabrication of these modified cylinders has necessitated the construction of X-ray orientation devices. The polished cylinders were protected with a plastic coating during X-ray orientation, grinding, and polishing of the flat surfaces. Attention is being given to methods of handling during microscopic inspection and ultimately during delivery of these precise parts. This part of the program is nearing completion.

D. Election Diffraction

A number of thin film deposits of β-SiC have been submitted for electron diffraction analysis. These epitaxially grown films were produced on various substrates and under a variety of experimental conditions at AFCRL. The samples were analyzed by electron diffraction to determine the degree of crystallinity and for purposes of phase identification. Some detailes on the methods follow below.

The specimen surface is placed almost parallel to the electron beam in reflection electron diffraction. The electrons diffract from surface asperities and are recorded on a film placed below the specimen. Amorphous materials yield concentric ring patterns and single crystal materials yield spot patterns. The ring patterns can be utilized to determine interplanar spacings of the reflecting planes, allowing specimen identification. In cases of large

crystallites (>10 microns), the rings tend to be spotty; for very small crystallites (<0.1 microns) or strained materials, the rings become diffuse.

Single crystal spot patterns can be utilized to determine the structural identification and orientation of the specimen. Particular orientations are achieved by rotations about two orthogonal axes normal to the electron beam and by a lateral translation. The camera constant (i.e., the relation between ring radius or spot distance from film center to interplaner spacing) is determined by means of a known standard. This constant is strongly dependent on electron beam voltage, but is rather insensitive to other microscope adjustments. All analyses reported herein have been carried out on a Hitachi HU-11 Electron Microscope operated at 100kv.

E. Chemical Analyses

Wet chemical and spectrochemical analyses were carried out on several specimens with the result shown in Table 4 below:

TABLE 4

CHEMICAL ANALYSES

Job No.	Spec. No.	Chemistry	Results
04106	RB-1	$RbIO_3$	I = 62.5 $^{w}/o$ Rb = 13.7 $^{w}/o$ HIO = 57.4 $^{w}/o$
04159	JS-Zr	Zirconium	C = 1.85 $^{w}/o$ N = 0.079 $^{w}/o$ H = 0.026 $^{w}/o$ S = none Si = 0.014 $^{w}/0$ Zr = 92.04 $^{w}/o$
04491		Cadmium Telluride	Cd = 43.9 $^{w}/o$ Te = 56.1 $^{w}/o$

F. Microhardness Measurements

Microhardness meausrements were performed utilizing a Leitz Microhardness Tester with Vickers or Knoop indentor and a 15gm load. An average hardness value based on five readings was obtained for various salt crystals and the results are summarized below:

TABLE 5

MICROHARDNESS MEASUREMENTS

Job No.	Spec. No.	Material	VHN	KHN
03008	LQ-33	KCl	10.6	
	LQ-46		10.0	
	LQ-54	KCl + KBr	20.8	
	LQ-45		21.0	
03012	LQ-13	RbCl		7.69
	LQ-53	RbCl		8.10
	LQ-53	RbCl		8.34
	LQ-70	KBr		8.62
	LQ-87	KCl		9.82
	LQ-87A	KCl		10.36
	LQ-88	KBr + KCl		19.75
	LQ-91	KCl +KOH		10.39

*Annealed